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OPTIMIZED SYNTHESIS AND CHARACTERIZATION OF CARBOXYMETHYLCELLULOSE DERIVED FROM SUGARCANE BAGASSE LIGNOCELLULOSE (*Saccharum officinarum* L.): COMPREHENSIVE FTIR AND QUALITY STANDARD ANALYSES

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ABSTRACT

Bagasse is a fibrous waste material derived from sugarcane (*Saccharum officinarum* L.) that can be useful in producing carboxymethyl cellulose (CMC). This study synthesized CMC from bagasse using NaOH concentrations of 25%, 35%, and 45%, along with 20% trichloroacetic acid (TCA), to produce high-quality CMC. The results revealed that the CMC synthesized with 35% NaOH exhibited properties closely aligning with the standards reported by the Food and Agriculture Organization (FAO). The drying loss, moisture content, pH, viscosity, NaCl content, purity, and degree of substitution were 3.1%, 5.247%, 6.91, 688.3 cP, 0.25%, 99.74%, and 1.08, respectively. Fourier-transform infrared analysis confirmed the functional groups characteristic of CMC, with prominent peaks at 3121.9 cm^{-1} (OH group), 2886.5 cm^{-1} (C-H group), 1663.6 cm^{-1} (COO^- group), and 1029 cm^{-1} (-O- group). These findings suggest that bagasse can be effectively utilized to produce high-quality CMC and potentially convert waste into value-added products.

Keywords: Carboxymethyl Cellulose, Bagasse, Synthesis, Characterization, Lignocellulose.

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INTRODUCTION

Recent advancements in biopolymer-based materials have spurred intensive exploration of lignocellulosic biomass as a sustainable alternative to cellulose.[1,2] Sugarcane bagasse (*Saccharum officinarum* L.), a major agro-industrial byproduct, contains a lignocellulosic matrix composed of cellulose (40–50%), hemicellulose (25–35%), and lignin (15–25%) in a complex, interlinked structure stabilized by covalent and non-covalent bonds.[3,4] This unique composition makes it an ideal substrate for carboxymethyl cellulose (CMC) production through modified processes, including selective alkaline delignification and controlled etherification.[5,6] This study holds high urgency in the context of sustainable biomass-based material development, given that sugarcane bagasse, an abundant agro-industrial residue, remains underutilized, while global demand for CMC continues to rise for pharmaceutical, food, and industrial applications.[6,7] The novelty of this work lies in its holistic approach to converting sugarcane bagasse lignocellulose into high-value CMC through selective delignification and controlled carboxymethylation, followed by comprehensive characterization using deconvoluted FTIR spectroscopy to map carboxymethyl group distribution and stringent pharmacopeial quality analysis. Key innovations include optimized reaction

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conditions to achieve a degree of substitution (DS) >0.8 with high purity,[8,9] along with an in-depth investigation of the relationship between native lignocellulose structure and the functional properties of synthesized CMC, a gap rarely addressed in prior studies.[10] These findings are anticipated to not only provide a circular economy-driven solution for lignocellulosic waste valorization but also establish a new benchmark for eco-friendly CMC production with commercial-grade quality.[2,7] This study introduces several key advancements compared to prior research on lignocellulose-based CMC synthesis. While earlier work predominantly relied on purified cellulose sources such as cotton,[11,12] our approach utilizes whole sugarcane bagasse lignocellulose—an underexplored yet highly abundant feedstock.[13] Previous syntheses often lacked systematic optimization of critical reaction parameters (e.g., NaOH concentration, temperature, and duration),[14] whereas our methodology rigorously refines these conditions to achieve superior yield and degree of substitution (DS). Beyond conventional FTIR characterization for functional group identification,[15] we implement a comprehensive analytical framework incorporating pharmacopeial-grade quality assessments (FAO standards) for viscosity, purity, and solubility.[16,17] This dual approach provides robust validation of CMC performance, a significant departure from earlier studies that prioritized synthesis over stringent quality verification. By bridging this gap, our work not only enhances reproducibility but also aligns with industrial requirements for high-grade CMC production. This study aims to optimize the synthesis of CMC from sugarcane bagasse lignocellulosic matrix by modifying key reaction parameters (NaOH concentration, temperature, and reaction time). It further conducts in-depth characterization of the CMC product using FTIR spectroscopy to verify successful carboxymethyl group substitution and evaluates critical quality parameters, including degree of substitution (DS), viscosity, and purity, against FAO standards. The ultimate goal is to produce a competitive functional material from renewable resources.

EXPRERIMENTAL

Materials

The sugarcane bagasse used in this research comes from sugarcane farms in Kediri, East Java., Indonesia. The materials was used in this researched are pro analysis Sodium Hydroxide pellets EMPLURA® (Millipore, KGaA, Germany), Toluene EMSURE® ACS (Millipore, KGaA, Germany), Aquadest grade II (PT. SMARTLAB, Smart-Lab, Indonesia), 95% Ethanol EMSURE® ACS (Millipore, KGaA, Germany), Isopropanol EMSURE® ACS (Millipore, KGaA, Germany), Trichloroacetic Acid EMSURE® ACS (Millipore, KGaA, Germany), Glacial Acetic Acid EMPARTA® ACS (Millipore, KGaA, Germany), Sodium Chlorite (Millipore, KGaA, Germany), Silver Nitrate EMSURE® ACS (Millipore, KGaA, Germany), 70% Methanol EMSURE® ACS (Millipore, KGaA, Germany) and Potassium Chromate EMSURE® ACS (Millipore, KGaA, Germany).

Extraction of Cellulose

Bagasse powder was first pulverized and then extracted using the Soxhlet extraction method as a dewaxing process, employing a 2:1 toluene-ethanol solution (300 ml) for 3.5 hours at a temperature of 100°C. Subsequently, hemicellulose was removed by treating the sample with 17.5% NaOH for 4 hours at 100°C. The resulting powder was then utilized for further processing. The next step involved reacting the powder with 100 mL of 1% $NaClO_2$ for 3 hours in an acidic medium at a temperature of 40°C for the bleaching process. This treatment was repeated two or more times until discoloration occurred in the sample, after which it was filtered and washed with distilled water until no acid remained. The purified cellulose fiber was then dried at a temperature of 30°C for 1 hour. Testing was conducted using organoleptic tests, drying shrinkage tests, moisture content tests, and FTIR analysis against cellulose to ensure that the results of the extraction were in the form of cellulose.[18]

Synthesis of CMC

5 grams of cellulose bagasse were weighed to create CMC. Next, 100 mL of isopropanol was added to the reaction mixture. The next step in the alkalinization process involved adding 20 mL of NaOH at concentrations of 25%, 35%, and 45%. The mixture was then heated on a hotplate for one hour at 30°C while stirring with a magnetic stirrer set at 200 rpm. Afterward, 20 mL of 20% trichloroacetic acid (TCA) was incorporated into the mixture. The mixture was then heated for four hours on a hotplate set at 45°C.

Filter paper was used to filter the hot mixture, and the residues were collected and steeped in 100 mL of 70% methanol. Glacial acetic acid was then used to neutralize the solution to pH 7. Once the solution reached a pH of 7, it was placed in a beaker covered with aluminum foil and left for a full day. After 24 hours, the solution was filtered, and the residue was dried for one hour at 60°C. The CMC derived from bagasse was then examined, along with tests for FTIR spectrum, moisture content, pH, viscosity, degree of substitution, and loss on drying.[18,19]

Characterization of CMC

Moisture Balance

The water content was measured within the CMC. The presence of water was attributed to the hygroscopic nature of activated carbon.[20] Samples weighing 0.5 g were combined and fed into the Karl Fischer instrument to provide the necessary measurements.[20]

Loss of Drying

A moisture balance tool was used to complete the inspection. To operate the device, turn it on, set the timer for 15 minutes, and adjust the temperature to 105°C. Wait until the word "ready" appears. A total of 1 g of CMC was placed in a sample plate, closed, and then the results were awaited.[20]

pH

1 g of CMC was dissolved in 100 mL of aqueous solution and heated to 60°C while stirring until fully dissolved. The pH was then measured.[20]

Viscosity

The viscosity of the formulation was measured using a First Touch Viscometer with spindle R-4. A total of 1 g of CMC powder was weighed, dissolved in 100 mL of water, and heated to 60°C while stirring until the powder was fully dissolved. The solution was then cooled to room temperature, and the viscosity was measured.[20]

Degree of Substitution

The degree of substitution of carboxylic groups in CMC is characterized as the average number of hydroxyl groups within the cellulose structure that have been substituted by carboxymethyl groups.[12,20]

Sodium Chloride Content

1 g of dry weight was weighed, and an Erlenmeyer flask was filled with CMC and diluted with 100 mL of Aquadest. After that, this solution was titrated using 5% K_2CrO_4 indicator and $AgNO_3$ 0.1 N.[12,21]

$$\text{NaCl (\%)} = \frac{0.584 \text{ x N } AgNO_3 \text{ x volume } AgNO_3}{\text{dry weight sampel (g)}}$$

Purity

The purity of CMC was calculated in the following method:[21]

$$\text{Purity} = 100\% - \%\ \text{NaCl}$$

FT-IR Analysis of CMC

The FTIR study was conducted using a Perkin Elmer 1600 spectrometer, which has a working range of 4000 cm^{-1} to 500 cm^{-1}. Using dry KBr, the sample was formed into a transparent pellet, and transmission mode spectra were acquired. Samples of cellulose and CMC made from sugarcane bagasse and cationic cellulose were analyzed.[4] The presence of the COO–, CH_2–, and –O– groups in the IR spectrum confirmed that the substitution reaction occurred.[22]

RESULTS AND DISCUSSION

Powdered bagasse was treated with NaOH to eliminate hemicelluloses and lignin. The essential chemical reagent that dissolves lignin and hemicellulose while leaving cellulose undissolved is NaOH (Fig.-1).[23] A 17.5% NaOH pretreatment broke the intramolecular hydrogen bonds in the powdered bagasse, altering the bonding network and strength.[18] The alkali-pretreated fibers were then bleached with 1% acidified sodium chlorite to further reduce the hemicellulose and lignin content, as these components could not be completely removed.[19] Approximately 64.88% of the bagasse from sugarcane was converted to cellulose. To produce high-quality cellulose derivatives, a high content of α-cellulose is necessary.[24] Reduced α-cellulose

concentration suggests an abundance of low-molecular-mass oligosaccharides, which could potentially impact the yield and quality of cellulose derivatives.[25,26] To generate CMC, cellulose was alkalized with 25%, 35%, and 40% (w/v) NaOH in the presence of isopropyl alcohol. This was followed by a carboxymethylation procedure using trichloroacetic acid (TCA). The purpose of employing NaOH as a reagent was to elongate the cellulose chains and facilitate the replacement of hydroxyl groups in the cellulose units with sodium carboxymethyl groups.[27]

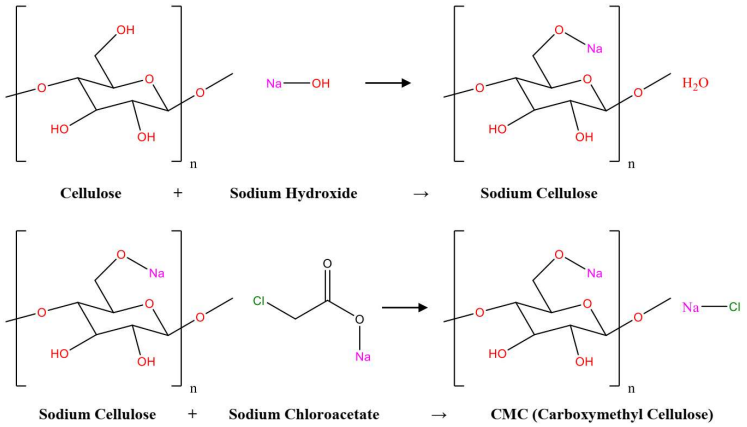

Fig.-1: Reaction Mechanism of CMC Formation

The analytical data of NaOH solutions (25%, 35%, and 45%) confirm full compliance with FAO standards, establishing their suitability for CMC synthesis in Table-1. The high purity (>99.5%) and low NaCl content (<0.5%) ensure minimal interference with cellulose etherification,[11,28] while the stable pH range (6.0-8.5) provides optimal alkalization conditions.[18,23] Although the 45% NaOH solution shows increased moisture loss (6.76±0.776%), this remains well below the FAO threshold (<12%), preserving reaction stability.[16,29] While rising viscosity (624.2-741.06 cP) with concentration could theoretically affect reagent diffusivity, the consistent degree of substitution (DS 1.08-1.10) demonstrates effective carboxymethyl group incorporation. This confirms sufficient reaction activation energy is maintained, attributable to sugarcane bagasse's favorable solubility in alkaline media.[20]

Table-1: Results of CMC Characteristics with NaOH Variations.

Parameters	NaOH Concentration			FAO Requirements [16]
	25%	35%	45%	
Color and odor	Odorless	Slight alkaline	Slight alkaline	White-cream
Solubility	Easily soluble	Easily soluble	Easily soluble	Easily soluble
Water content	2.939±1.072	5.247±0.436	5.303±0.193	<12%
Loss on drying	3.23±1.470	3.1±0.436	6.76±0.776	<12%
pH	6.62±0.037	6.91±0.037	7.11±0.024	6.0-8.5%
% NaCl	0.23±0.009	0.25±0.020	0.31±0.029	<0.5%
Purity	99.76±0.009	99.74±0.020	99.69±0.029	>99.5%
Viscosity	624.2±16.671	688.3±3.920	741.06±22.488	>25 Cps
Degree of substitution	1.08	1.08	1.10	0.2-1.5%

Notably, higher NaOH concentrations (45%) didn't significantly enhance DS values compared to 25-35% solutions, suggesting rate-limiting factors in hydroxyl group accessibility or monochloroacetate reaction kinetics. The FAO-compliant purity and viscosity profiles reinforce sugarcane bagasse's viability as a sustainable industrial CMC precursor.[29,30] For commercial-scale production, 35% NaOH emerges as the optimal concentration, offering balanced processing economics through moderate viscosity (688.3 cP) and the lowest moisture loss (3.1%). Further characterization using FTIR and SEM/XRD would provide valuable insights into how NaOH concentration affects the structural integrity and thermal stability of derived CMC products in Fig.-2 and Table-2.

The combined results of FAO-compliant NaOH characterization (25%, 35%, 45%) and FTIR analysis provide comprehensive insights into the reagent quality-CMC synthesis efficiency relationship. The NaOH

parameters (purity >99.5%, NaCl <0.5%, pH 6.0-8.5) meeting FAO standards ensure optimal reaction conditions for carboxymethylation.[15,28,31]

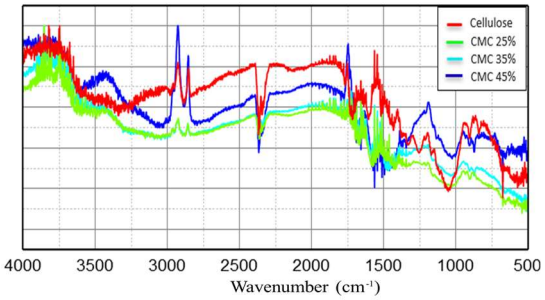

Fig.-2: Spectrum of FTIR of CMC

This is evidenced by the emergence of characteristic C=O peaks at 1664-1683 cm^{-1} in all CMC FTIR spectra, confirming successful carboxylate group formation. The most intense peak at 1682.96 cm^{-1} in 45% NaOH-derived CMC correlates with the highest DS value (1.10), demonstrating that high NaOH purity (99.69±0.029%) enhances carboxymethyl substitution efficiency despite increased solution viscosity (741.06 cP).[3,6,26]

Table-2: The FTIR Spectra of Cellulose and CMC with different Amounts of NaOH

Wavenumber (cm^{-1})				Assignment
Cellulose	CMC (w/v of NaOH 25%)	CMC (w/v of NaOH 35%)	CMC (w/v of NaOH 45%)	
3363.03	3187.5	3121.9	3608.96	OH stretching
2880.81	2889.4	2886.5	2882.73	CH stretching
-	1664.6	1663.6	1682.96	Region C=O (shown as CMC)
1047.39	1023.2	1029	1040.64	C-O-C asymmetrical stretching of the bridge

Significant modifications in the O-H stretching region (3121.9-3608.96 cm^{-1}) indicate transformation of cellulose's hydrogen bonding network post-alkalization. The peak shift to higher wavenumbers in 45% NaOH-CMC (3608.96 cm^{-1}) versus native cellulose (3363.03 cm^{-1}) reflects reduced intramolecular hydrogen bonding due to -CH_2COOH group substitution. Meanwhile, maintained C-O-C peaks (1040-1047 cm^{-1}) verify the preservation of cellulose's fundamental backbone structure. These findings reinforce the FAO characterization results, showing that while 45% NaOH had higher moisture loss (6.76±0.776%), it didn't compromise CMC quality, as confirmed by DS >1 and characteristic FTIR spectra.[12,26] The collective data establish NaOH-modified (35-45%) sugarcane bagasse as a sustainable industrial CMC precursor, with 35% NaOH recommended as the optimal balance between reaction efficiency and product stability.

CONCLUSION

This research successfully developed an optimized method for producing carboxymethylcellulose (CMC) from sugarcane bagasse, focusing on NaOH concentration as the key reaction parameter. The study demonstrated that a 35% NaOH concentration produced CMC with the most favorable combination of properties: appropriate viscosity (688.3 cP), good substitution degree (DS 1.08), and full compliance with FAO standards for purity (>99.5%) and salt content (<0.5%). Infrared spectroscopy analysis clearly showed the expected chemical changes, with new absorption peaks confirming successful modification of the cellulose structure while maintaining its core framework. The work establishes sugarcane agricultural waste as a practical raw material for making quality CMC, offering an environmentally friendly alternative to conventional sources. These findings provide manufacturers with a reliable approach for producing bio-based CMC that meets international specifications for various industrial applications.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-3: Good Health and Well-being*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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